Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

ALKALI REDUCTION OF LAKE ST. JOE IRON ORE CONCENTRATE

J.M.D. Wilson
Yu-Cong Ma
Mineral Processing Laboratory

September 1981

This document is the unedited internal report prepared primarily for discussion and internal reporting purposes. It does not represent a final expression of the opinion of the Canada Centre for Mineral and Energy Technology (CANMET)

Ce document est un rapport préliminaire non-révisé et rédigé principalement pour fin de discussion et de documentation interne. Il ne représente nullement l'expression définitive de l'opinion du Centre canadien de la technologie des minéraux et de l'énergie (CANMET)

Project MRP-3.4.0.0.03
Iron Ore Processing

JAN 20 1984
MICROMEDIA

DECLASSIFIED
DATE 5/6/83
AUTHORIZED BY

1977

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 81-5 (CF)

CONFIDENTIAL

ALKALI REDUCTION OF LAKE ST. JOE IRON ORE CONCENTRATE

by

J.M.D. Wilson* and Yu Cong Ma**

ABSTRACT

Acid leaching tests show that the alkali ratio (SiO_2/K_2O) of Lake St. Joe pilot plant flotation concentrate can be raised from 50:1 to 99:1 with .5N H_2SO_4 or HCl at 80°C over 2 hours. Silica losses are relatively small but concurrent weight losses are 4% and 4.6% respectively. Oxalic acid leaching and H_2SO_4 pressure leaching showed poorer results.

---

*Physical Scientist, Mineral Processing Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.

**Visiting Research Mining Engineer, People's Republic of China.

INTRODUCTION

The Lake St. Joseph iron ore property is located on the south shore of Lake St. Joseph, approximately 190 miles north of Thunder Bay, Ontario and is owned jointly by Stelco, Algoma and Dofasco. Magnetite is the predominate iron mineral and average drill core assays are 24% soluble Fe with 49% SiO_2 (1). One of the technical problems hindering the development of Lake St. Joe is that the ore contains appreciable quantities of alkalis, potassium and sodium, contained in the gangue minerals which are mainly phlogopite, K-feldspar and Na-plagioclase. Phlogopite is the major source of the alkalis and is present in major amounts, >10%, according to XRD studies (2).

It is now well known that alkalis in the modern blast furnace contribute to a wide range of operational problems resulting in increased production costs (3). Although several methods are known for removing alkalis in the blast furnace a better approach, from an operational point of view, is to reduce the alkali levels in the pellets before charging to the blast furnace. To date this is not economically feasible due to the extra capital investment and operating costs and decreased iron recovery.

A constraint which must be placed on any proposed upgrading step is that the silica level of the concentrate be maintained at 2.9% to satisfy present blast furnace practice. As well, iron losses must be minimized. Alkalis are removed from the blast furnace mainly by means of the slag and their removal is inversely proportional to slag basicity which depends on the amount of silica charged. Thus for efficient alkali removal it has been calculated that the weight ratio of silica to alkali in the blast furnace feed must be maintained at a minimum level of 100:1 (SiO_2/K_2O) (4). The alkali levels are expressed solely in terms of K_2O because it can be analyzed quickly by means of XRF whereas Na_2O cannot and because the Na_2O content of

blast furnace materials can be empirically related to the K_2O content anyway. Also it is felt that potassium presents the more serious problem because it is more difficult to remove in the slag (5). Therefore, if the silica level is specified at 2.9% then the K_2O must be reduced to 0.029%.

Pilot-plant testwork using flotation beneficiation on the Lake St. Joe ore has produced a rougher concentrate. A report of previous work done on this material showed that several beneficiation methods had been tried which were attrition scrubbing, electrostatic separation, calcium chloride roasting, concentrate blending with silica, flotation with HF and sulphuric acid leaching. The first three showed no appreciable improvement. Blending achieved the desired result but was rejected due to higher capital and operating costs. Flotation with HF produced a concentrate alkali ratio of 60:1 with a 3.3% weight loss but the harsh operating conditions mitigated against this method. Sulphuric acid leaching raised the alkali ratio to 55:1 with a weight loss of only 0.8%.

This report covers a part of the work done on alkali removal in the Iron Ore Processing project whose objective is to develop technology for the removal of alkalis from Ontario iron ores to render them more acceptable to the steel industry.

METHODS AND RESULTS

CANMET bagan work on this concentrate in April 1981 when 50 lb of Lake St. Joe rougher concentrate was received via Dofasco. The objective was to obtain an alkali ratio of 100:1 while maintaining SiO_2 at 2.9% with minimal iron losses. The material as received was sized and sent for chemical and mineralogical analysis. The results are summarized in Tables 1 and 2. An examination of Table 1 shows that the alkali ratio has fallen short of the 100:1 objective and further upgrading is required. Analytical accuracies are

±.005%. The conclusions of the mineralogist's report indicate that (6):

1. Magnetite liberation increases with fineness of sample. In the -200 +270 M fraction 88 wt% of the magnetite is free while 98.7% is free in the -500 M fraction. Most of the unliberated magnetite is attached to quartz.
2. The K content is due to phlogopite, K-feldspar and mafic minerals. Phlogopite is attached to magnetite whereas the K-feldspar is attached to quartz and mafic minerals. In the -400 M sizes, some of both phlogopite and K-feldspar are present as free grains.

There were too few K-bearing mineral grains to obtain a quantitative analysis of K liberation.

Due to the fineness of the concentrate as received (57% -500 M) it was decided that a promising beneficiation method might be acid leaching. Three acids were chosen for this study H_2SO_4, HCl and $H_2C_2O_4.2H_2O$ using two acid levels, two leaching times and three temperatures. It is known that oxalic acid easily forms complexes with aluminum and therefore it was used in an attempt to break-up and form a complex with the alumino silicate structures of phlogopite and alkali feldspars.

The method of leaching was simple. A 50 g sample of the concentrate was placed in a 400 ml beaker and then 200 ml of acid solution was added. The mixture was heated on an electric hot plate and stirred constantly with an impeller. After leaching the contents were filtered twice using 300 ml of distilled water then dried, weighed and assayed. Results are shown in Tables 3 to 5 and plotted in Fig. 1 to 5. A further series of pressure leach tests were tried using .1N H_2SO_4. The autoclave contained 100 g of concentrate and 400 ml of acid in a one litre vessel. The residue was treated as before. Results are shown in Table 6 and Fig. 6.

DISCUSSION

The pressure leach tests were disappointing. Although temperatures of only 80°C were used it was felt that further tests were not indicated, keeping in mind the high capital and operating costs necessary to implement an autoclave system.

The oxalic acid leach proved non-selective and weight losses were high for little improvement in the alkali ratio.

HCl and H_2SO_4 gave comparable results. Both produced alkali ratios of 99:1 with a weight loss of over 4% at 0.5N. The tests indicated that both acid strength and temperature are important while time is not as important after the initial 120 minutes.

Analytical accuracies are such that for an alkali ratio of 100:1 the accuracy is ± 15-20%.

H_2SO_4 seems to remove less silica than does HCl as is shown on comparison of Tables 3 and 4 for 0.5N acids.

The leach results show promise and although the weight losses are high, future optimization of the system should reduce this. The mineralogy report indicates that a regrind of the +400 M material would liberate more phlogopite and K-feldspar grains for leaching. Leaching at a higher % solids should be tried. Also some of the Fe loss could be recovered by precipitation.

CONCLUSIONS

1. There are two main alkali-bearing minerals, phlogopite and K-feldspar roughly present in the ratio of 2:1 respectively. Phlogopite is associated with the magnetite while the K-feldspar is with the quartz.
2. Pressure leaching with sulphuric acid did not raise the alkali level sufficiently and was abandoned.

3. Oxalic acid leaching did not raise the alkali level sufficiently either.
4. The target of a 100:1 alkali ratio at 2.9% SiO_2 is attainable but with 4% iron losses using 0.5N HCl or 0.5N H_2SO_4.

RECOMMENDATIONS

Further work should be continued to optimize the leach system and to reduce the weight loss.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the assistance of S. Sastri for his many helpful ideas and suggestions and for the use of the pressure leaching equipment. Thanks is also extended to G. Leigh for help with the pressure leaching tests and to W. Feader for his assistance with the computer graphics.

REFERENCES

1. Taylor, B.W., Lamb, L.J. and Jones, F.R. "Lake St. Joseph - iron ore prospect to superconcentrate project"; Bull. Can. Inst. Min. Metall.; March 1972.
2. Dofasco, unpublished report.
3. Davies, J., Moon, J.T. and Traice, F.B. "Alkalis in the blast furnace"; Ironmaking Steelmaking; 4:151-161; 1978.
4. Lu, W.K. Personal communication.
5. Ashton, J.D., Gladysz, C.V., Walker, G.H. and Holditch, J. "Alkali control at Dofasco"; in Proc. of Symp. on Alkalis in Blast Furnaces; edited by Standish and Lu; 1973.
6. Petruk, W., Owens, D. and Pinard, R.G. "Mineralogical and image analyses study of a magnetite concentrate from the Lake St. Joe iron deposit"; Division Report MRP/MSL 81-4 (CF); CANMET, Energy, Mines and Resources Canada; 1981.

TABLE 1

Chemical Analysis of Lake St. Joe concentrate by size fraction

Size Fraction	Wt %	Tot Fe%	Sol Fe%	Na_2O%	SiO_2%	K_2O%	Ratio
Head	100.0	69.53	68.91	.05	3.01	.06	50
+200 M	0.4	*	*	.14	10.74	.16	67
-200 +270 M	5.6	64.89	64.79	.11	8.67	.12	72
-270 +400 M	13.6	68.70	68.29	.06	3.91	.07	56
-400 +500 M	23.3	69.84	69.32	.04	2.75	.06	46
-500 M	57.1	70.25	69.94	.03	1.90	.05	38

*Insufficient sample

TABLE 2

Mineralogical Analysis of Lake St. Joe Concentrate by Size Fraction

Mineral	Head	+200M	-200 +270M	-270 +400M	-400 +500M	-500M
Magnetite -free	86.3	79.0	77.3	91.1	93.9	92.7
-Unliberated	4.3	9.6	10.7	2.7	1.8	1.2
Hematite	0.4	0.2	0.4	0.1	0.1	0.3
Quartz	8.5	8.1	8.4	3.7	3.4	5.3
K-spar +phlogopite	.08	2.7*	0.7	1.0	0.4	0.1
Phlogopite	na	na	0.4	na	0.3	na
K-spar	na	na	0.3	na	0.1	na
Plagioclase	nd	nd	0.8	0.3	0.2	.02
Mafics	0.4	0.4	1.4	1.1	0.2	0.4

na not analyzed
nd not detected
* mostly K-spar

TABLE 3

H_2SO_4 Leach

Normality	Reaction Temp(^{o}C)	Reaction Time(min.)	Wt. Loss %	Tot Fe%	SiO_2%	K_2O%	Ratio
.1	25	45	.2	69.9	3.00	.055	55
	25	120	.2	70.0	2.89	.048	60
	50	45	.4	69.9	2.86	.042	68
	50	120	.2	69.8	2.86	.039	73
	80	45	1.0	70.2	2.88	.041	70
	80	120	.6	70.3	2.79	.039	72
.5	25	45	1.8	69.7	2.96	.042	71
	25	120	1.8	69.9	2.84	.042	68
	50	45	2.5	69.7	2.83	.038	74
	50	120	4.4	69.9	2.84	.033	86
	80	45	2.7	70.0	2.82	.035	81
	80	120	4.0	69.9	2.87	.029	99

TABLE 4

HCl Leach

Normality	Reaction Temp(°C)	Reaction Time (min.)	Wt. Loss %	Tot Fe%	SiO_2%	K_2O%	Ratio
	25	45	.2	69.0	2.78	.047	59
	25	120	.2	69.2	2.93	.050	59
.1	50	45	1.6	69.8	2.81	.039	72
	50	120	.5	69.2	2.78	.043	65
	80	45	.6	70.3	2.82	.038	74
	80	120	2.2	69.0	2.81	.038	74
	25	45	1.2	70.0	2.68	.037	72
	25	120	1.6	68.6	2.77	.039	71
.5	50	45	1.0	70.0	2.82	.036	78
	50	120	2.6	70.0	2.58	.032	81
	80	45	3.4	69.9	2.51	.031	81
	80	120	4.6	70.0	2.66	.027	99

TABLE 5

$H_2C_2O_4 \cdot 2H_2O$ Leach*

Normality	Reaction Temp(°C)	Reaction Time (Min.)	Wt. Loss %	Tot Fe%	SiO_2%	K_2O%	Ratio
.1	25	45	2.2	69.6	2.78	.049	57
	25	120	1.9	70.1	3.05	.050	61
	50	45	2.0	70.1	3.08	.050	62
	50	120	2.3	70.1	3.08	.050	62
	50	420	2.1	70.1	3.04	.050	61
.5	25	45	2.8	69.7	2.81	.049	57
	25	120	2.6	69.5	2.85	.049	58
	50	45	2.1	69.7	2.69	.044	61
	50	120	4.4	69.5	2.74	.040	69

*Oxalic decomposes at 60°C.

TABLE 6

.1N H_2SO_4 Pressure Leach

Reaction Pressure (lbs/in^2)	Reaction Temp(^oC)	Reaction Time(Hours)	Wt. Loss %	Tot Fe%	SiO_2%	K_2O%	Ratio
100	40	3	1.4	70.3	2.98	.041	73
100	40	7	.5	69.9	2.87	.040	72
100	80	3	1.4	70.2	2.74	.040	69
100	80	7	1.5	69.9	2.60	.040	65
250	40	3	1.7	69.9	2.90	.042	69
250	40	7	1.0	70.0	2.86	.040	72
250	80	3	.8	70.0	3.16	.040	79
250	80	7	.9	70.0	2.89	.040	72

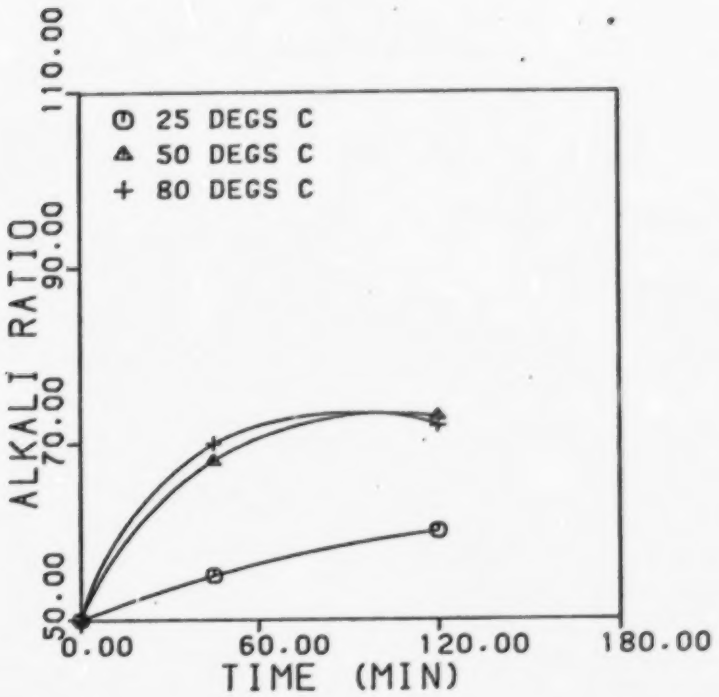

FIG. 1 .1N H2SO4 LEACH

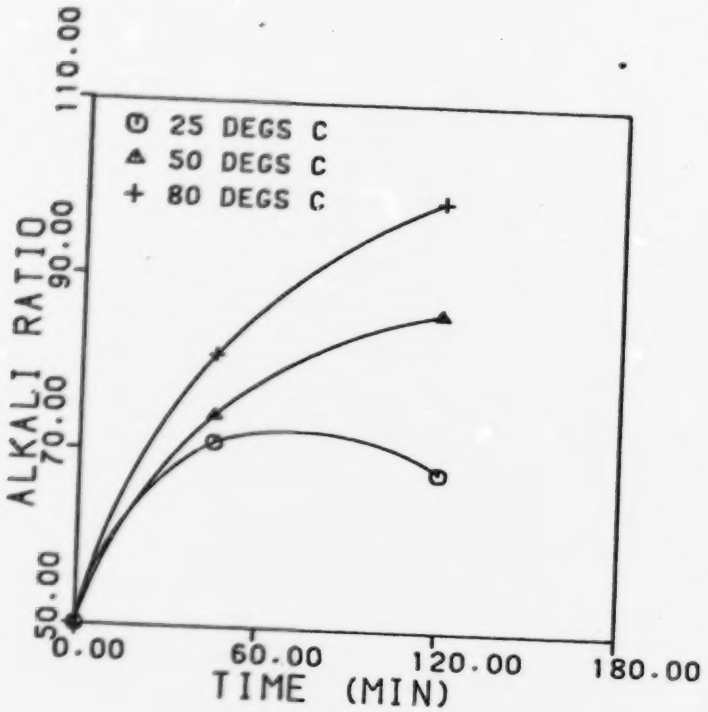

FIG. 2 .5N H2SO4 LEACH

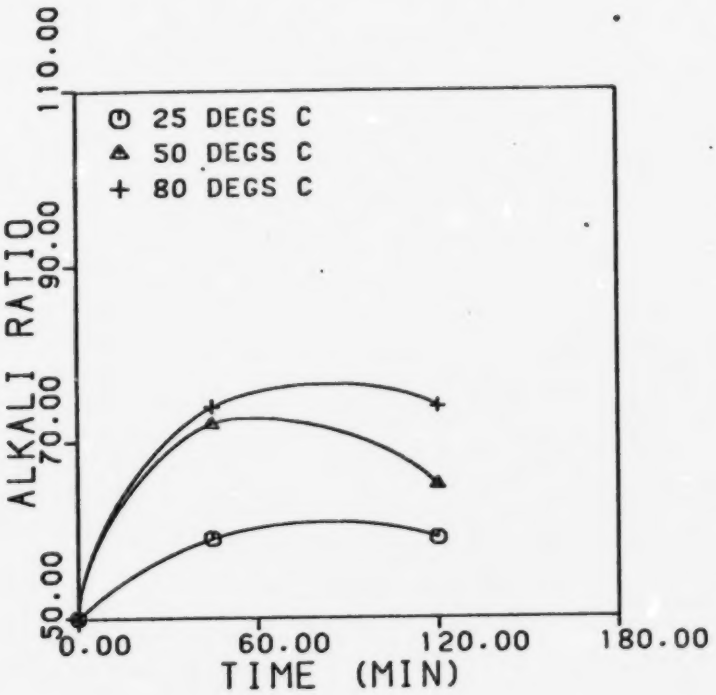

FIG. 3 .1N HCL LEACH

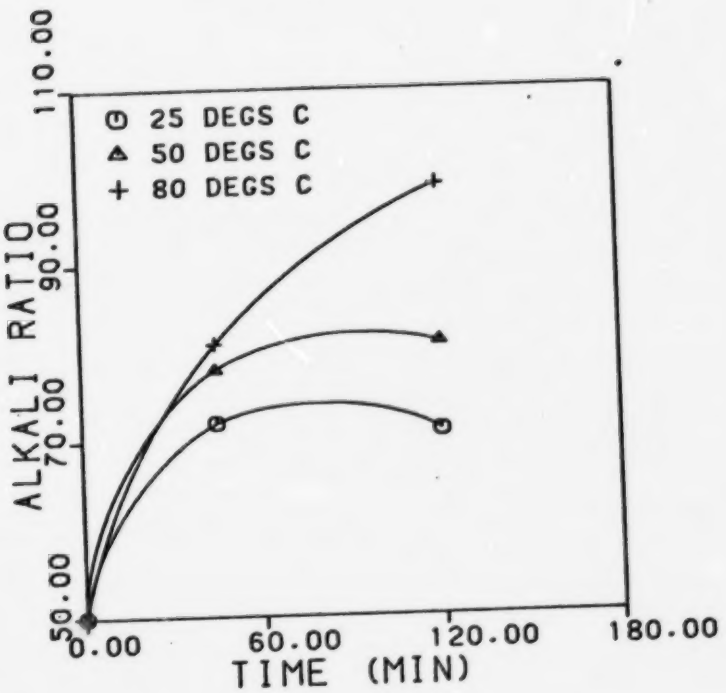

FIG. 4 .5N HCL LEACH

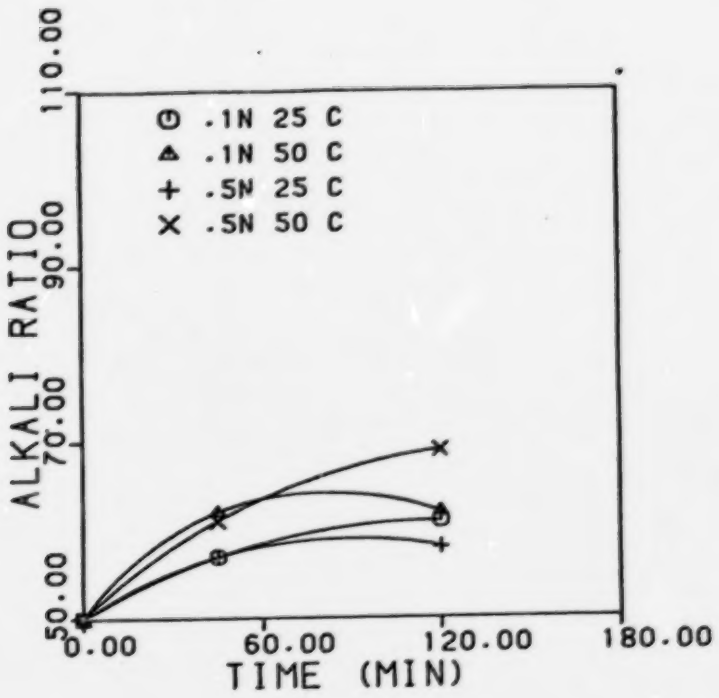

FIG. 5 OXALIC ACID LEACH

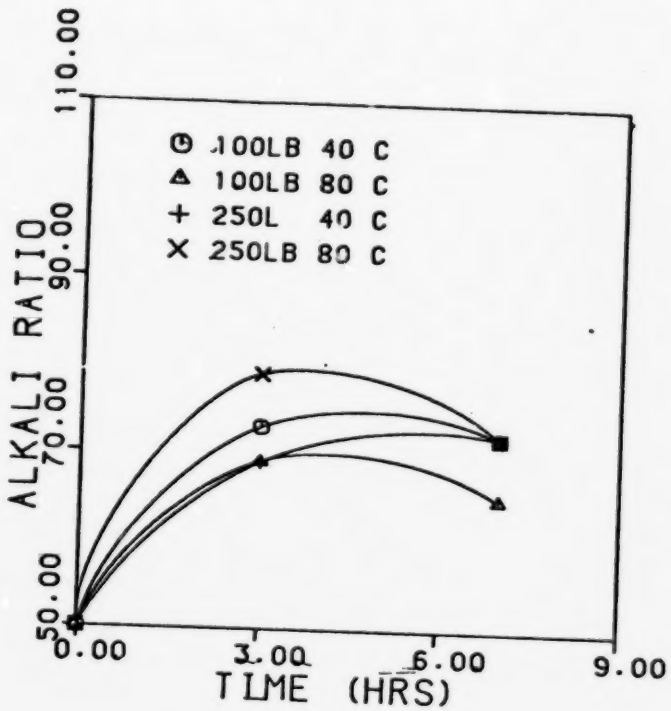

FIG. 6 .1N H2SO4 PRESSURE LEACH